

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of

Rinze BENEDICTUS et al.

Serial No.: 10/642,507

Filed: August 18, 2003

For: HIGH DAMAGE TOLERANT Al-Cu ALLOY



CLAIM FOR PRIORITY

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

The benefit of the filing date of the following prior foreign application filed in the following foreign country is hereby requested for the above-identified application and the priority provided in 35 USC 119 is hereby claimed:

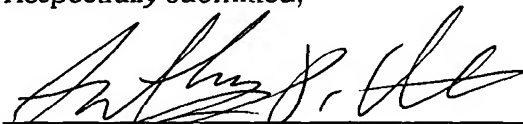
European Patent Application No. 02078443.5 filed on August 20, 2002.

In support of this claim, a certified copy of said original foreign application already in English is filed herewith.

It is requested that the file of this application be marked to indicate that the requirements of 35 USC 119 have been fulfilled and that the Patent and Trademark Office kindly acknowledge receipt of this document.

Respectfully submitted,

Date: Sept 26, 2003

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Patentanmeldung Nr. Patent application No. Demande de brevet n°

02078443.5

Der Präsident des Europäischen Patentamts;
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets
p.o.

R C van Dijk



Anmeldung Nr:
Application no.: 02078443.5
Demande no:

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Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.
If no title is shown please refer to the description.
Si aucun titre n'est indiqué se referer à la description.)

High damage tolerant Al-Cu alloy

In Anspruch genommene Priorität(en) / Priority(ies) claimed /Priorité(s)
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High damage tolerant Al-Cu alloy

The present invention relates to a high damage tolerant Al-Cu alloy having a high toughness and an improved fatigue crack growth resistance while maintaining good strength levels according to claim 1, a method for producing a rolled high damage tolerant Al-Cu alloy product having a high toughness and an improved fatigue crack growth resistance according to claim 8 and a rolled alloy sheet product for aeronautical applications. More specifically, the present invention relates to a high damage tolerant Al-Cu-Mg alloy designated by the Aluminium Association ("AA") 2000-series for structural aeronautical applications with improved properties such as fatigue crack growth resistance, strength and fracture toughness. The invention also relates to a rolled alloy product which is suited to be used as fuselage skin or lower wing skin of an aircraft.

It is known in the art to use heat treatable aluminium alloys in a number of applications involving relatively high strength such as aircraft fuselages, vehicular members and other applications. Aluminium alloys 2024, 2324 and 2524 are well known heat treatable aluminium alloys which have useful strength and toughness properties in T3, T39 and T351 tempers.

The design of a commercial aircraft requires various properties for different types of structures on the aircraft. Especially for fuselage skin or lower wing skin it is necessary to have properties such as good resistance to crack propagation either in the form of fracture toughness or fatigue crack growth. At the same time the strength of the alloy should not be reduced. A rolled alloy product either used as a sheet or as a plate with an improved damage tolerance will improve the safety of the passengers, will reduce the weight of the

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aircraft and thereby improve the fuel economy which translates to a longer flight range, lower costs and less frequent maintenance intervals.

It is known in the art to have 2x24 alloy compositions with the following chemistry, in weight percent:

Cu: 3.7 - 4.4
Mg: 1.2 - 1.8
Mn: 0.15 - 0.9
10 Cr: 0.05 - 0.10
Si: ≤ 0.50
Fe: ≤ 0.50
Zn: ≤ 0.25
Ti: ≤ 0.15

15 the balance aluminium and incidental impurities.

US-5,593,516 discloses a high damage tolerant Al-Cu alloy with a balanced chemistry comprising essentially the following composition (in weight %):

Cu: 2.5 - 5.5
20 Mg: 0.1 - 2.3
Cu_{max}: - 0.91 Mg + 5.59
Cu_{min}: - 0.91 Mg + 4.59
Zr: up to 0.2, or
Mn: up to 0.8

25 balance aluminium and unavoidable impurities. It also discloses T6 and T8 tempers of such alloys which gives high strength to a rolled product made of such alloy.

US-5,897,720 discloses a high damage tolerant Al-Cu alloy with a „2024“-chemistry comprising essentially the following composition (in weight %):

30 Cu: 3.8 - 4.9
Mg: 1.2 - 1.8
Mn: 0.3 - 0.9

the balance aluminium and unavoidable impurities wherein
35 the alloy is annealed after hot rolling at a temperature

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at which the intermetallics do not substantially dissolve. The annealing temperature is between 398°C and 455°C.

US-5,938,867 discloses a high damage tolerant Al-Cu alloy with a „2024“-chemistry comprising essentially the following composition (in weight %):

Cu: 3.8 - 4.9

Mg: 1.2 - 1.8

Mn: 0.3 - 0.9

balance aluminium and unavoidable impurities wherein the ingot is inter-annealed after hot rolling with an anneal temperature of between 385°C and 468°C.

European patent EP-0473122 as well as US-5,213,639 disclose an aluminium base alloy comprising essentially the following composition (in weight %):

Cu: 3.8 - 4.5

Mg: 1.2 - 1.8

Mn: 0.3 - 0.9

Fe: ≤ 0.12

Si: ≤ 0.10

the remainder aluminium, incidental elements and impurities, wherein such aluminium base is hot rolled, heated and again hot rolled, thereby obtaining good combinations of strength together with high fracture toughness and a low fatigue crack growth rate. More specifically, US-5,213,639 discloses an inter-anneal treatment after hot rolling the cast ingot with a temperature between 479°C and 524°C and again hot rolling the inter-annealed alloy wherein the alloy contains one or more elements from the group consisting of

Cr: 0.02 - 0.40

V: 0.01 - 0.5

Hf: 0.01 - 0.40

Cr: 0.01 - 0.20

Ag: 0.01 - 1.00

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Sc: 0.01 - 0.50.

Such alloy shows a 5% improvement over the above mentioned conventional 2024-alloy in T-L fracture toughness and an improved fatigue crack growth resistance at certain ΔK -levels.

EP-1170394-A2 discloses an aluminium sheet product with improved fatigue crack growth resistance having an anisotropic microstructure defined by grains having an average length to width aspect ratio of greater than about 4 and comprising essentially the following composition, (in weight %):

Cu: 3.5 - 4.5

Mg: 0.6 - 1.6

Mn: 0.3 - 0.7

15 Zr: 0.08 - 0.13,

the remainder substantially aluminium, incidental elements and impurities. The examples show a Zr-level in the range of 0.10 to 0.12 while maintaining an Mg-level of more than 1.30. Such alloy has an improvement in compressive yield strength properties which is achieved by respective sheet products in comparison with conventional 2524-sheet products. Furthermore, the strength and toughness combinations of such sheet products with high Mn variants have been described better than those of 2524-T3. Throughout the high anisotropy in grain structure the fatigue crack growth resistance could be improved.

Furthermore, it is described that low copper-high manganese samples exhibited higher properties than high copper-low manganese samples. Results from tensile strength measurements showed that high manganese variants exhibited higher strength values than the low manganese variants. The strengthening effect of manganese was reported to be surprisingly higher than that of copper.

It is the object of the present invention to provide

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a high damage tolerant 2024-alloy having a high toughness and an improved fatigue crack growth resistance while maintaining good strength levels of conventional 2024, 2324 or 2524 alloys. It is a further
5 object of the present invention to provide aluminium alloy sheet products having an improved fracture toughness and resistance to fatigue crack growth for aircraft applications such as fuselage skin or lower-wing skin.

10 Yet a further object of the present invention is to provide rolled aluminium alloy sheet products and a method for producing those products so as to provide structural members for aircrafts which have an increased resistance to fatigue crack growth and to provide an
15 improved fracture toughness while still maintaining high levels of strength.

More specifically, there is a general requirement for rolled 2000-series aluminium alloys within the range of 2024 and 2524 alloys when used for aeronautical
20 applications that the fatigue crack growth rate ("FCGR") should not be greater than a defined maximum. An FCGR which meets the requirements of high damage tolerance 2024-series alloy products is e.g. FCGR below 0.001 mm/cycles at $\Delta K = 20 \text{ MPa}\sqrt{\text{m}}$ and 0.01 mm/cycles at $\Delta K = 40$
25 $\text{MPa}\sqrt{\text{m}}$.

The present invention solves the above mentioned objects by the features of independent claims 1, 8 and 12. Preferred embodiments are characterized within the sub-claims wherein an aircraft fuselage sheet or an
30 aircraft lower-wing member sheet is defined in claim 16.

In accordance with the invention there is disclosed a high damage tolerant Al-Cu alloy having a high toughness and an improved fatigue crack growth resistance by maintaining high levels of strength which comprises
35 essentially the following composition (in weight %):

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Cu: 3.8 - 4.7

Mg: 1.0 - 1.6

Zr: 0.06 - 0.18

Mn: ≤ 0.50 and Mn: > 0 , preferably > 0.15 5 Fe: ≤ 0.15 Si: ≤ 0.15 ,

and Mn-containing dispersoids, the balance essentially aluminium and incidental elements and impurities, wherein the Mn-containing dispersoids are at least
10 partially replaced by Zr-containing dispersoids.

It has surprisingly been found that lower levels of manganese result in a high toughness and an improved fatigue crack growth resistance specifically in areas where the toughness and fatigue crack growth resistance
15 under tensile load are critical. The alloy of the instant invention in the T3 temper has significant improved high damage tolerance properties by lowering the amount of manganese and by partially replacing manganese-containing dispersoids by zirconium containing
20 dispersoids. At the same time it is vital to carefully control the chemistry of the alloy.

The main improvement of the alloy according to the present invention is an improved fatigue crack growth resistance at the lower ΔK -values which leads to
25 significant longer lifetimes. The balance of high damage tolerance properties and mechanical properties of the alloy of the present invention is better than the balance of conventional 2024 or 2524-T3 alloys. At the same time the toughness levels are equal or better to
30 2524 alloy levels.

The alloy of the present invention is either unrecrystallized or recrystallized. However, it has been found that the high damage tolerance properties such as fracture toughness or strength may be further improved
35 by adding zirconium.

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The amount (in weight %) of manganese is preferably in a range of 0.20 to 0.45%, most preferably in a range of 0.25 to 0.30%. Mn contributes to or aids in grain size control during operations. The preferred levels of manganese are lower than those conventionally used in conventional 2x24 alloys while still resulting in sufficient strength and improved damage tolerance properties. In order to optimise the improved high damage tolerance properties the chemical composition of the alloy of the present invention preferably meets the proviso that $Zr \geq 0.09$ when $Mn \leq 0.45$ and $Cu \geq 4.0$.

The amount (in weight %) of copper is in a range of 4.0 to 4.4, preferably in a range of 4.1 to 4.3. Copper is an important element for adding strength to the alloy. It has been found that a copper content of 4.1 or 4.2 results in a good compromise in strength, toughness, formability and corrosion performance while still resulting in sufficient damage tolerance properties.

The preferred amount (in weight %) of magnesium is in a range of 1.0 to 1.4, most preferably in a range of 1.1 to 1.3. Magnesium provides also strength to the alloy product.

The preferred amount (in weight %) of zirconium is in a range of 0.09 to 0.15 thereby partially replacing Mn-containing dispersoids. The balance of manganese and zirconium influences the recrystallisation behaviour. Throughout the addition of zirconium more elongated grains may be obtained which also results in an improved fatigue crack growth resistance. Zirconium may also be at least partially replaced by chromium wherein $[Zr] + [Cr] \leq 0.20$. Preferred amounts (in weight %) of chromium and zirconium are in a range of 0.05 to 0.15, preferably in a range of 0.10 to 0.13. The balance of zirconium and chromium as well as the partial replacement of Mn-containing dispersoids and Zr-containing dispersoids

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result in an improved recrystallisation behaviour and more elongated grains.

A preferred alloy composition of the present invention comprises essentially the following
5 composition (in weight %):

Cu: 4.0 - 4.2

Mn: 0.20 - 0.65

Mg: 1.0 - 1.3.

Another preferred alloy according to the present
10 invention comprises essentially the following composition (in weight %):

Cu: 4.0 - 4.2

Mg: about 1.2

Zr: 0.10 - 0.15

15 Mn: 0.20 - 0.65

Fe: ≤ 0.10

Si: ≤ 0.10 .

Even more preferred, an alloy according to the present invention comprises essentially the following
20 composition (in weight %):

Cu: 4.1 or 4.2

Mg: about 1.2

Zr: about 0.14

Mn: 0.20 - 0.65

25 Fe: ≤ 0.10

Si: ≤ 0.10 .

The balance is aluminium and inevitable impurities and incidental elements. Typically, each impurity element is present at 0.05% maximum and the total of impurities is
30 0.20% maximum. The best results are achieved when the alloy rolled products have a recrystallised micro-structure meaning that 75% or more, and preferably more than 80% of the grains in a T3 temper are recrystallised.

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The alloy according to the present invention may further comprise one or more of the elements Zn, Ag, Hf, V, Sc, Ti or Li, the total amount less than 1.00 (in weight %). These additional elements may be added to
5 further improve the balance of the chemistry and enhance the forming of dispersoids.

A method for producing a rolled high damage tolerant Al-Cu alloy product having a composition as set out above and having a high toughness and an improved
10 fatigue crack growth resistance according to the invention comprises the steps of

a) casting an ingot with the following composition (in weight percent):

Cu: 3.8 - 4.7
15 Mg: 1.0 - 1.6
Zr: 0.06 - 0.18
Mn: ≤ 0.50 and Mn: > 0 , preferably > 0.15
Fe: ≤ 0.15
Si: ≤ 0.15 ,

20 and Mn-containing dispersoids, the balance essentially aluminium and incidental elements and impurities, wherein the Mn-containing dispersoids are at least partially replaced by Zr-containing dispersoids,

25 b) homogenising and/or pre-heating the ingot after casting,
c) hot rolling the ingot and optionally cold rolling into a rolled product,
d) solution heat treating,
30 e) quenching the heat treated product,
f) stretching the quenched product, and
g) naturally ageing the rolled and heat-treated product.

After hot rolling the ingot it is possible to anneal and/or re-heat the hot rolled ingot and again hot
35 rolling the rolled ingot. It is believed that such r -

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heating or annealing enhances the fatigue crack growth resistance by producing elongated grains which - if recrystallised - maintain a high level of toughness and good strength. It is furthermore possible to conduct a surface heat treatment between hot rolling and cold rolling at the same temperatures and times as during homogenisation, e.g. 1 to 5 hours at 460°C and about 24 hours at 490°C. The hot rolled ingot is preferably inter-annealed before and/or during cold rolling to further enhance the ordering of the grains. Such inter-annealing is preferably done at a gauge of app. 4.0 mm for one hour at 350°C. Furthermore, it is advisable to stretch the rolled and heat-treated product in a range of 1 to 5%, preferably in a range of 1 to 3%, and then naturally aging the stretched product for more than 5 days, preferably about 10 to 15 days.

The present invention provides a high damage tolerant rolled Al-Cu alloy sheet product which has high toughness and an improved fatigue crack growth resistance with the above described alloy composition which is preferably produced in accordance with the above described method. Such rolled alloy sheet product has preferably a gauge of around 2.0 mm to 12 mm for applications such as fuselage skin and about 25 mm to 50 mm for applications such as lower-wing skin. The present invention thereby provides an aircraft fuselage sheet or an aircraft lower-wing member sheet with improved high damage tolerance properties.

The foregoing and other features and advantages of the alloy according to the invention will become readily apparent from the following detailed description of preferred embodiments. Some of the enhanced high damage tolerant properties are shown in the appended drawings:

Fig. 1 shows the fatigue crack growth properties versus a 2524 reference alloy,

Fig. 2 Kahn-tear versus yield strength properties

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compared to 2024-T351 commercially available alloys and 2024-T351 pure alloys, and

Fig. 3 Kahn-tear versus yield strength properties as shown in Fig. 2 but in average L-T and T-L direction.

5 On an industrial scale 8 different aluminium alloys have been cast into ingots having the following chemical composition as set out in Table 1.

10 Table 1: Chemical composition of the DC-cast aluminium alloys, in weight %, Si about 0.05%, Fe about 0.06%, balance aluminium and inevitable impurities.

Alloy	Alloying Element				
	Cu	Mn	Mg	Zr	Cr
AA2024	4.4	0.59	1.5	0	0
AA2524	4.3	0.51	1.4	0	0
1	4.4	0.40	1.3	0.06	0
2	4.3	0.41	1.3	0.09	0
3	4.2	0.61	1.2	0.10	0
4	4.2	0.43	1.2	0.14	0
5	4.1	0.31	1.2	0.14	0
6	4.1	0.21	1.2	0.14	0
7	4.4	0.21	1.4	0.10	0
8	4.4	0.21	1.3	0	0.08

15 The alloys have been processed to a 2.0 mm sheet in the T351 temper. The cast ingots were homogenized at app. 490°C, then hot rolled at app. 410°C. The plates were further cold rolled, surface heat treated and stretched by app. 1%. All alloys have been tested at least after 10 days of natural aging.

20 Then the ultimate tensile strength properties and the unit propagation energy as well as the Kahn-tear was measured in the L and T-L direction. The testing was done in accordance with ASTM B871 (1996) for the Kahn tear tests, and EN-10.002 for the tensile tests.

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Tabl 2: Tensile properties and toughness of alloys 1 to 8 of table 1 in the L and T-L direction.

Alloy	L		T-L	
	PS (MPa)	UTS (MPa)	UPE (kJ/m ²)	TS/Rp
AA2024	344	465	162	1.74
AA2524	338	447	331	1.99
1	324	441	355	1.92
2	335	446	294	1.95
3	335	445	338	2.04
4	338	449	322	2.02
5	337	449	335	1.98
6	320	419	335	1.98
7	332	442	266	1.91
8	337	449	289	1.92

5 As identified in Table 2 and shown in Fig. 2 and 3
the Kahn-tear versus yield strength properties of the
alloys according to the present invention are better
than those of conventional 2024-T351 in commercially
available form or pure form. Furthermore, the minimum
10 level of manganese is in between 0.21 and 0.31 while at
a level of 0.21 the strength level is still good.

In order to identify the fatigue crack growth rate
(FCGR) all alloys were tested according to ASTM E-647 on
80 mm wide M(T) panels at $R = 0.1$ at constant load and a
15 frequency of 8 Hz. The lifetime as shown in Table 3 is
defined as the time (in number of cycles) that the crack
grows from a length of 5 mm to 20 mm. The maximum stress
was 54 MPa. The initial notch was 4.1 mm. Anti-buckling
device are not used. The results were shown in table 3
20 and Fig. 1.

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Table 3: Fatigue crack growth rate with ΔK -level is $\text{MPa}\sqrt{\text{m}}$ for all alloys compared with commercially available AA2024 alloy (= baseline).

Alloy	Cycles between $a=5$ and 20mm	Improvement in lifetime over AA2024
AA2024	163830	baseline
AA2524	216598	32%
1	338468	107%
3	517215	216%
4	526866	222%
6	416750	154%
7	272034	66%
8	284609	74%

5 From the results of Table 3 and Fig. 1 it is clear that the preferred amount of Mn is in a range of 0.25 to 0.45 (in weight %) and the preferred range of Zr is in between 0.09 and 0.15 (in weight %). Copper is preferably present in an amount below 4.3 and magnesium
10 is preferably present in an amount below 1.3 (in weight %).

From the results of Table 3 and according to Fig. 1 (Region A) it can be seen that alloys 3, 4 and 6 have a significantly improved lifetime over conventional AA2024
15 alloys preferably at ΔK -levels in a range of 5 to 15 $\text{MPa}\sqrt{\text{m}}$. Hence, the fatigue crack growth resistance at those lower ΔK -values results in significant longer lifetimes of the alloy and enhances its usefulness for aeronautical applications.

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Claims

1. High damage tolerant Al-Cu alloy having a high toughness and an improved fatigue crack growth resistance, comprising essentially the following composition (in weight percent):
5 Cu: 3.8 - 4.7
 Mg: 1.0 - 1.6
 Zr: 0.06 - 0.18
10 Mn: ≤ 0.50 and Mn: > 0 , preferably > 0.15
 Fe: ≤ 0.15
 Si: ≤ 0.15 ,
 and Mn-containing dispersoids, the balance essentially aluminium and incidental elements and
15 impurities, wherein the Mn-containing dispersoids are at least partially replaced by Zr-containing dispersoids.
2. Alloy as claimed in claim 1, wherein said alloy is
20 recrystallised to at least 75%.
3. Alloy as claimed in claims 1 or 2, wherein the amount (in weight %) of Mn is in a range of 0.20 to 0.45.
- 25 4. Alloy as claimed in one of the preceding claims, wherein the amount (in weight %) of Cu is in a range of 4.0 to 4.4, preferably in a range of 4.1 to 4.3.
- 30 5. Alloy as claimed in one of the preceding claims, wherein the amount (in weight %) of Mg is in a range of 1.0 to 1.4, preferably in a range of 1.1 to 1.3.
- 35 6. Alloy as claimed in one of the preceding claims, wherein the amount (in weight %) of Zr is in a range of 0.09 to 0.15.

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7. Alloy as claimed in one of the preceding claims,
wherein said alloy further comprises one or more of
5 the elements Zn, Ag, Hf, V, Sc, Ti or Li, the total
amount less than 1.00 (in weight %).
8. A method for producing a rolled high damage tolerant
Al-Cu alloy product according to any one of the
10 preceding claims and having a high toughness and an
improved fatigue crack growth resistance, comprising
the steps of
- a) casting an ingot with the following composition
in weight percent):
- 15 Cu: 3.8 - 4.7
Mg: 1.0 - 1.6
Zr: 0.06 - 0.18
Mn: ≤ 0.50 and Mn: > 0 , preferably > 0.15
Fe: ≤ 0.15
20 Si: ≤ 0.15 ,
and Mn-containing dispersoids, the balance
essentially aluminium and incidental elements
and impurities, wherein the Mn-containing
dispersoids are at least partially replaced by
25 Zr-containing dispersoids,
- b) homogenising and/or pre-heating the ingot after
casting,
- c) hot rolling the ingot and optionally cold
rolling into a rolled product,
- 30 d) solution heat treating,
e) quenching the heat treated product,
f) stretching the quenched product, and
g) naturally ageing the rolled and heat-treated
product.
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9. Method as claimed in claim 8, wherein after hot rolling the ingot, annealing and/or reheating the hot rolled ingot and again hot rolling the rolled ingot.
- 5 10. Method as claimed in claims 8 or 9, said hot rolled ingot is inter-annealed before and/or during cold rolling.
- 10 11. Method as claimed in any one of claims 8 to 10, wherein said rolled and heat-treated product is stretched by about 1 to 3 % and naturally aged for more than 5 days.
- 15 12. A high damage tolerant rolled Al-Cu alloy sheet product having a high toughness and an improved fatigue crack growth resistance with an alloy composition according to one of the claims 1 to 7 and/or produced in accordance with any one of the claims 8 to 11.
- 20 13. A rolled Al-Cu alloy sheet product according to claim 12, wherein said product is a structural member of an aircraft.
- 25 14. A rolled sheet product according to claim 12 or 13, wherein said product is a fuselage skin of an aircraft.
- 30 15. A rolled sheet product according to claim 12 or 13, wherein said product is a lower-wing member of an aircraft.
- 35 16. An aircraft fuselage sheet or an aircraft lower-wing member sheet produced from high damage tolerant rolled Al-Cu alloy sheet with an alloy composition according to any one of the claims 1 to 7 and/or

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produced in accordance with any one of the claims 8
to 11.

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Abstract

The present invention concerns a high damage tolerant Al-Cu alloy of the 2000 series having a high toughness
5 and an improved fatigue crack growth resistance, comprising essentially the following composition (in weight percent) Cu: 3.8 - 4.7, Mg: 1.0 - 1.6, Zr: 0.06 - 0.18, Mn: \leq 0.50 and Mn: $>$ 0, preferably $>$ 0.15, Fe: \leq 0.15, Si: \leq 0.15, and Mn-containing dispersoids, the
10 balance essentially aluminium and incidental elements and impurities, wherein the Mn-containing dispersoids are at least partially replaced by Zr-containing dispersoids. There is also disclosed a method for producing a rolled high damage tolerant Al-Cu alloy
15 product having a high toughness and an improved fatigue crack growth resistance, and applications of that product as a structural member of an aircraft.

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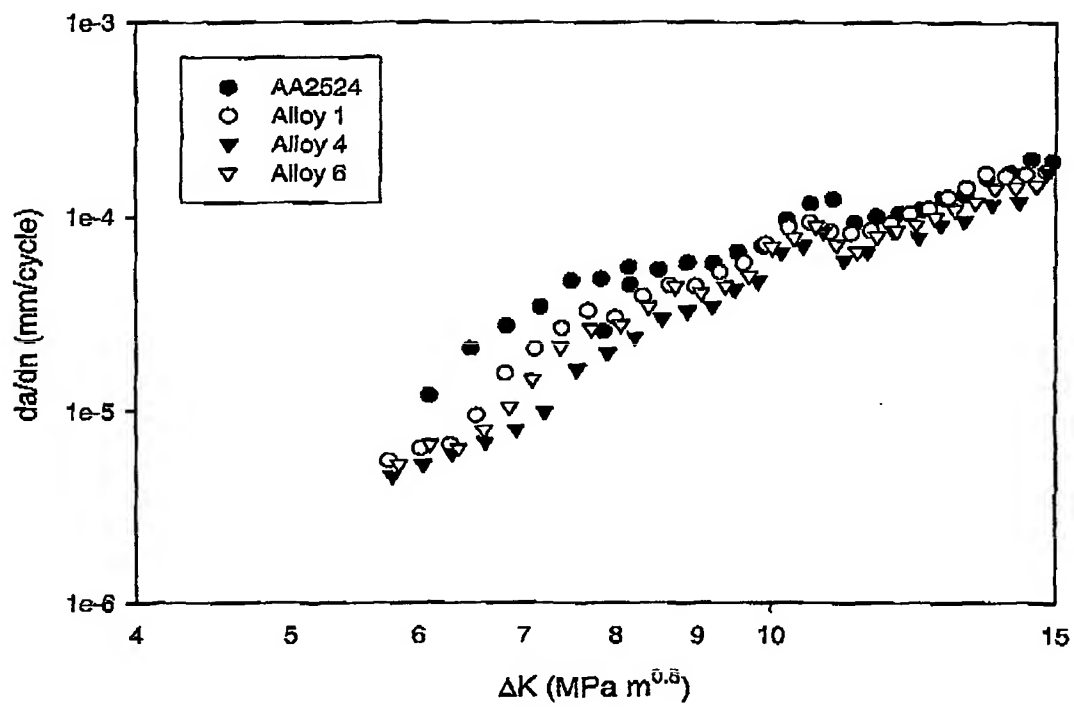


Fig. 1

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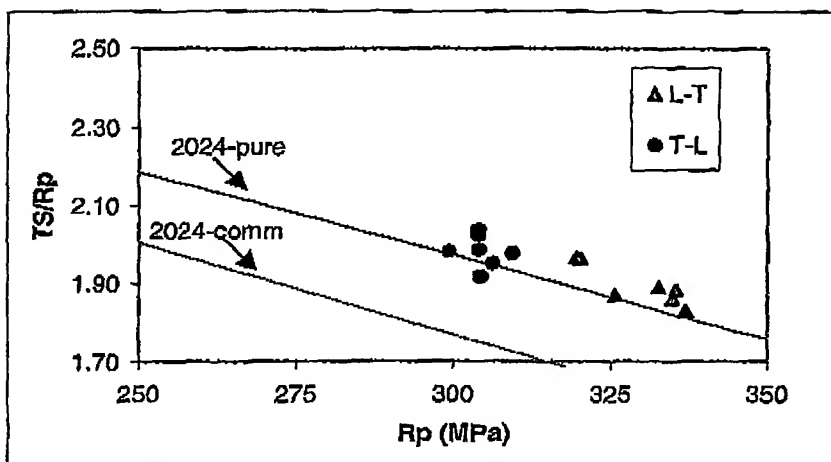


Fig. 2

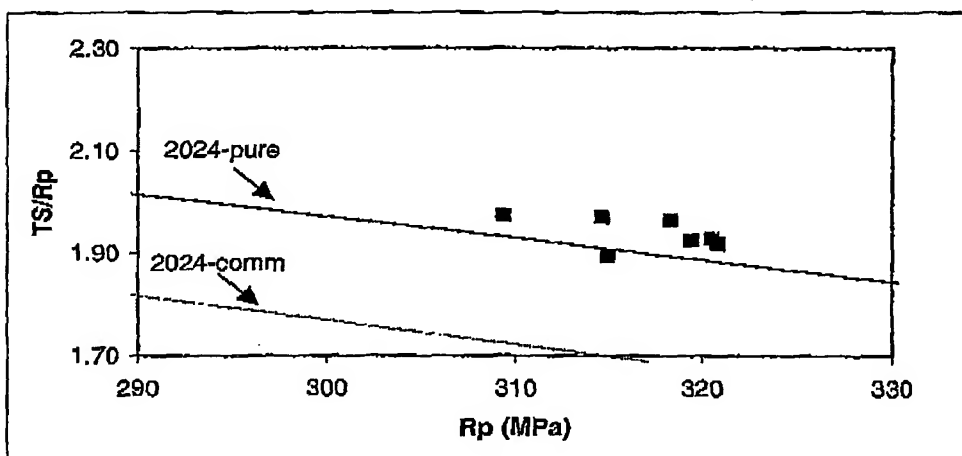


Fig. 3